Approved by BNC Managing Director_____Paul Lum_____Date:_____

Approved by PI (s) /Super User (s): _Peter Hosemann/Andy Minor_Date:_____

Standard Operating Procedure
BNC FEI Quanta 3D FEG/FIB ESEM
Version 2011 Aug 30

I. Purpose

This Standard Operating Procedure (SOP) outlines requirements to be considered by an authorized user of the FEI Quanta 3D FEG/FIB ESEM (FEG/FIB) as well as describes the normal operation of the FEG/FIB and any hazards that may be encountered during normal operation. Finally, the SOP explains how to minimize any hazards and how to respond in an emergency situation. This document is to be reviewed one year from the date of approval or as conditions warrant, whichever is the shorter time period.

II. Personnel

A. Authorized Personnel: The FEG/FIB may be operated only by authorized personnel who are fully cognizant of all safety issues involved in the operation of such a device. These personnel are to ensure that the FEG/FIB is only operated in the manner laid out in this document. To become an authorized user, one must:

1. Complete Environment, Health & Safety (EH&S) training class.
2. Take the baseline BNC Safety Orientation class
3. Read and fully understand the SOP
4. Receive training on the FEG/FIB by an authorized user.
5. Sign the authorized user sheet to affirm that the above steps have been completed.

B. Unauthorized personnel: No unauthorized personnel may enter the BNC clean room facility unless accompanied by an authorized user. All visitors must be briefed on the safety protocol and must wear appropriate protective eyewear located on the premises.

III. Hazards

A. Electrical Hazards: electrical shock or electrocution could result from direct contact with high voltage. Such hazards are typically interlocked by the FEG/FIB system. High voltage electrode and conductors are located inside the FEG/FIB system chassis. In addition, the power supply unit has connections behind the FEG/FIB chassis. Do not disconnect the external lines. Use normal precautions with external house (208/110VAC) connections behind the FEG/FIB chassis.

C. Chemical: Bottled gases are used with the FEG/FIB. Do not disconnect or tamper with gas lines on the FIB.

D. Pressure Hazards: Pressurized bottled and house gases are used with the FEG/FIB. Do not disconnect or tamper with gas lines behind the FEG/FIB. Contact PI or lab management for information.

E. Radiation is generated by the FEG as well as radioactive material may be utilized on this FEG/FIB. Use precautions. Reference Radiation RUA associated with FEG/FIB.

IV. Hazard Controls

A. Electrical

1. Enclosures for protection against the high voltages of the FEG/FIB may only be removed after the power supply has been unplugged from the outlets and after following the safety procedures outlined in the safety and operations manual provided by the manufacturer.

2. Only qualified personnel may perform all internal maintenance to the FEG/FIB.

3. Every portion of the electrical system, including the printed circuit cards, should be assumed to be at dangerous voltage level.

C. Chemical and Pressure

1. Enclosures for protection against valves and internal gas plumbing may only be removed after the system has been turned off and gases have been valve off and relieved of line pressure.

2. Only qualified personnel may perform all internal gas maintenance to the FEG/FIB.

D. Other

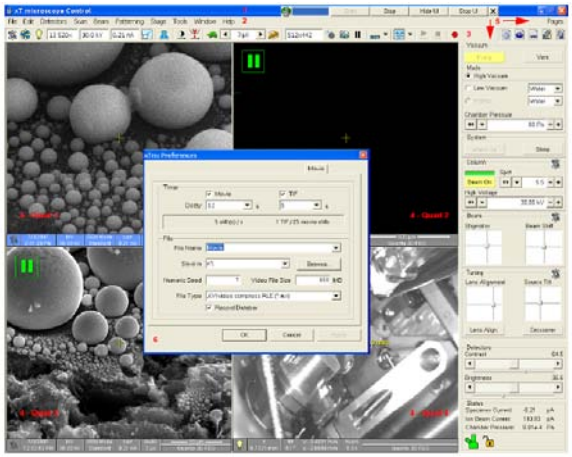
1. Proper eye protection must be worn at appropriate times in the clean room and as necessary while preparing samples.
2. Check logbook and radioactive material warning signs before using the system.

V. Normal Operation

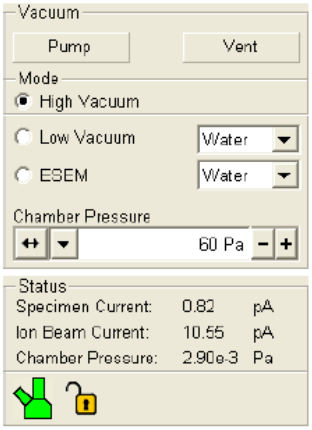
A. Inspect all electrical and water connections for damage and connectivity.
B. Complete the "check-in" log. Check if SEM chamber contains radioactive samples.
C. Log into the User Interface (UI) with the password for the users.

D. Caution: If you wish to place samples into the chamber, STOP Immediately. DO NOT OPEN THE CHAMBER IF A RADIOACTIVE SAMPLE IS IN THE CHAMBER!! See either Prof(s) Hosemann or Minor for permission or instructions.
E. If there are no radioactive samples in the chamber, you may proceed. Set the system control power button to ON (green).

F. Open up window XT Microscope Control



G. For venting the chamber, Click Vent. The vacuum will decrease allowing the chamber door to be opened. This will take approximately 5 minutes. There should be a small icon of the Quanta 3D FEG machine located on the bottom right hand corner of the screen. The chamber will be displayed in orange indicating the pump is not running. When chamber is fully vented place the new sample into the experimental chamber. Use the sample holders provided in the suitcase or desiccators.



H. Carefully place the sample into the slot. Make sure the sample is aligned so that it's evenly flat. Take the screw driver and screw the socket (located at the side).
I. Close the chamber door. Make sure the door is closed completely.
J. For Pumping the chamber. Click Pump. Allow approximately 5 minutes for the vacuum to reach base pressure. The chamber will now be displayed in green indicating that the pump is running.
K. If only SEM visualization is being operated, then turn on the electron beam ("Beam on" button in e-mode button).
L. If FIB and SEM are being operated, then turn on "Wake up" button. This turns all beams on.
M. Use focus, brightness and contrast and stig to get a good image in SEM.
N. Link the sample to the z-axis (Focal Point). (Blue button on the top)
O. Move the sample to the 10mm working distance. Watch the chamber camera such that you do not hit the pole piece.
P. If you want to operate the FIB, find the eucentric height with the tilting method.
Q. Proceed with Imaging.
R. When you have completed imaging, Click Vent. The vacuum will decrease allowing the chamber door to be opened. This will take approximately 5 minutes. There should be a small icon of the Quanta 3D FEG machine located on the bottom right hand corner of the screen. The chamber will be displayed in orange indicating the pump in not running.
S. Place your sample back into the proper container.
T. Close the chamber door. Make sure the door is closed completely.
U. Click Pump. Allow approximately 5 minutes for the vacuum to reach base pressure. The chamber will now be displayed in green indicating that the pump is running.

V. Just hit the "sleep button" next to the "wake up button". The system will turn itself off appropriately.
W. Log off the software and complete the logbook entry information.

CAUTION: Things to be aware of:

1. Do not hit the pole piece while using the stage!
2. Always work in eucentric height or further away. Never closer.
3. Only insert the GIS if you are in eucentric height.
4. Be careful, if the micromanipulator is installed in the chamber.
5. Never change the operation mode (high voltage, low vacuum, water vapor, etc…) without consulting either Prof (s) Hosemann or Minor.
6. Log in your time and beam status in the logbook. Report any unusual circumstances.

VI. Emergency Procedures

A. FEG/FIB accidents: Notify lab management and PI immediately.

B. In event you need to shutdown the system. Just hit the "sleep button" next to the "wake up button". The system will turn itself off appropriately.

C. Power outage: If there is a power outage, turn off the FEG/FIB per the FEG/FIB shut down procedure to avoid a hazardous situation when power is restored.

D. The following is reference material taken from the FEI Power Off Procedures. Refer to Page 23. Pages 20 and 21 of this SOP contains the full system shutdown procedure.

POWER OFF

Take sufficient measures to avoid power failures as much as possible. If it occurs while the instrument is completely operational, the microscope comes down to a safe state and the following happens:

- The HV is switched off abruptly.
- Both electron and ion emissions are switched off.
- The specimen chamber vents gently, automatically.
- The column isolating valves close to save the vacuum in the gun areas.

  Note:
  Electron gun IGP's are supported by the battery unit for several hours to keep sufficient vacuum for the source to operate.
- The momentary adjustments of all system parameters (accelerating voltage, magnification, stage positions) are lost if they were not saved.

Note:
If the power failures occur occasionally it is recommended to use the microscope Uninterruptible Power Supply (UPS).

The Emergency Off

is similar to that which would happen after a MAINS power off. Here are several possibilities how to quickly switch off the electrical power completely in case of emergency:

1. Push the red **EMERGENCY** (EMO) button (option - see the Safety Manual).

If the button is not installed proceed as follows:

2. Switch off the breaker switch labeled **MAINS S1** at the cabinet back, which is placed at the very right side in the row.

FIGURE 3-2 MAINS SWITCH BOARD

If this is not easily accessible:

3. Turn off the mains wall switch (if present), and / or disconnect the mains plug from the mains socket.

Caution!
If the Startup procedure fails (see above), contact the FEI Service Engineer.

VII. Authorized Users

I have read and understood the Standard Operating Procedures for FEG/FIB

Name (print)/contact information	Signature	Date	PI Initial

Appendix A – In case of medical emergencies, consult lab safety protocol or lab safety plan.

In the event of a FEG/FIB accident, follow the procedure below:

1. Ensure that the FEG/FIB is shut off per **VI. Emergency Procedures**.

2. Provide for the safety of the personnel (first aid, evacuation, etc.) as needed.

3. Obtain medical assistance for anyone who may be injured.

UC Optometry Clinic (Normal Hours)	642-2020
UC Optometry Clinic (24 Hour Emergencies)	642-0992
University Health Services (Emergency)	642-3188
Ambulance (urgent medical care)	9-911

4. If there is a fire, pull the alarm, and contact the fire department by calling 9-911. Do not fight the fire unless it is very small and you have been trained in fire fighting techniques.

5. Inform the Office of Environment Health, & Safety (EH&S) as soon as possible.

6. During normal working hours, call the following:

EH&S Office	642-3073
BNC Safety Officer (Paul Lum) 121 Stanley	666-3356
EH&S Health & Safety Manager	642-3073

After normal working hours, call 642-6760 to contact the UC Police Department who can contact the above using their emergency call list.

7. Inform ***(PI NAME)*** and the BNC safety officer as soon as possible. If there is an injury, ***(PI NAME)*** will need to submit a report of injury to the Worker's Compensation Office.

8 After the incident, do not resume use of the FEG/FIB system until the lab manager and EH&S has reviewed the incident and approved the resumption of research.

2 SYSTEM OVERVIEW

The Quanta 3D FEG is a combination of two systems:

- A Scanning Electron Microscope (SEM) produces enlarged images of a variety of specimens achieving magnification over 100 000× providing high resolution imaging in a digital format,
- A Focused Ion Beam (FIB) system is capable of fast and precise milling of the specimen material, revealing the structure under the surface layer, making cross sections, deposition layers, etc. The ion system produces high resolution images as well.

The integration of both systems yields a powerful analytical tool for obtaining any data from any sample in three dimensions.

This important and widely used analytical tool provides exceptional field of view, minimal specimen preparation, and the ability to combine the technique with X-ray microanalysis.

Users can switch between the two beams for quick and accurate navigation and milling. Convergence of the SEM and FIB at a short working distance allows precision "slice-and-view" cross-sectioning and analysis at high resolution. The instrument provides optimum throughput, resolution and automation.

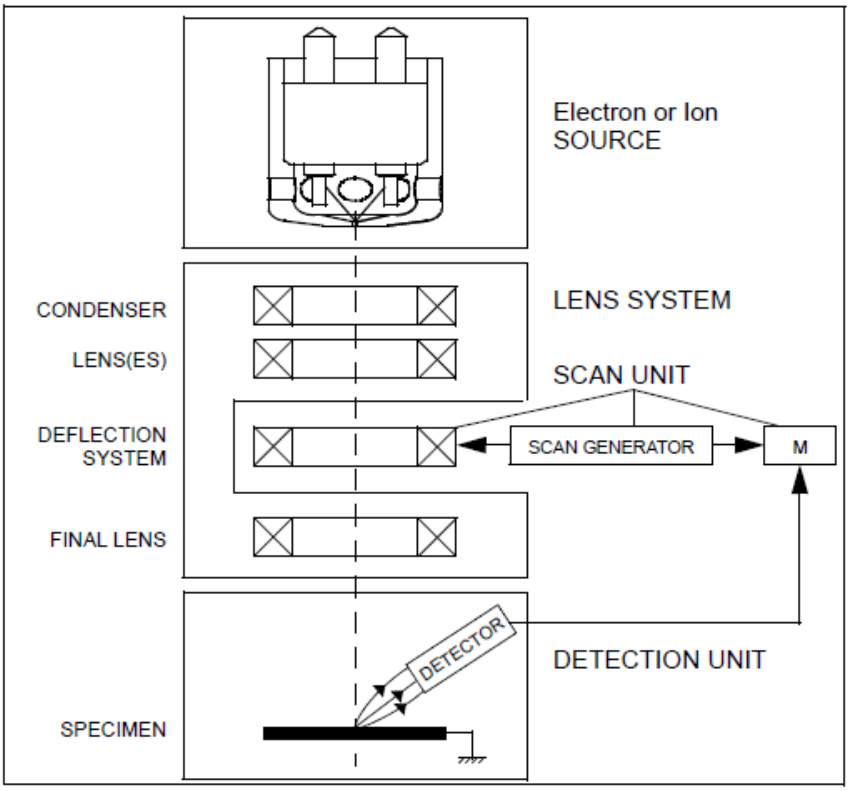
FIB/SEM instrument provide an expanded range of capabilities not possible with separate FIB and SEM tools:

- High-resolution electron beam images of FIB cross sections without eroding the feature of interest
- Real-time cross-section images and videos with the electron beam during FIB milling
- Focused electron beam charge neutralization during FIB milling
- High resolution elemental microanalysis of defect cross sections
- Imaging of sample surfaces with the electron beam during navigation without erosion or gallium implantation from the ion beam
- TEM sample preparation with *in situ* conductive coating

Main instrument components used for imaging of the samples are:

- **Electron / Ion source**
  The beam of electrons or ions (particles) is emitted within a small spatial volume with a small angular spread and selectable energy.
- **Lens system**
  The beam enters the lens system consisting of several electromagnetic or electrostatic lenses and exits to hit the specimen surface.
- **Scan unit**
  The scan generator signal, fed to the deflection systems, moves the beam in a raster pattern over the specimen area. The electrical voltage changes as it rasters. This signal, modulated by the detection system signal produces the onscreen imaging of the specimen surface.
- **Detection unit**
  Particles striking the specimen react with atoms of the sample surface in different manners:
  – The electron beam produces three basic types of signal: X-rays, electrons and photons.
  – The ion beam produces ions and electrons.
  The detector system picks up the particles or signals, converts them into an amplified electrical signal which is then sent to the control PC and displayed on the monitor.

FIGURE 2-1 COLUMN SCHEMATIC OVERVIEW



VACUUM SYSTEM

The entire electron path from gun to specimen must be under vacuum so that the particles do not collide with air molecules. The Quanta 3D FEG has the following operating vacuum modes to deal with different sample types:

- **High Vacuum** (HiVac)
- **Low Vacuum** (LoVac)
- **ESEM™**

Various levels of vacuum are necessary, so a Turbo Molecular Pump (TMP) backed by a scroll pre-vacuum pump (PVP), obtains the necessary specimen chamber pressure.

In the gaseous modes (LoVac, ESEM) the column is under lower pressure than the specimen chamber, where the pressure ranges from 10 to 2600 Pa (0.1 to 20 Torr), with auxiliary gas up to 4000 Pa (30 Torr). Either mode can use water vapours from a built-in water reservoir, or an auxiliary gas which is supplied by an user and connected to a gas inlet provided for this purpose. Observation of outgassing or highly charging materials can be made using one of these modes without the need to metal coat the sample, which would be necessary for conventional HiVac mode.

Specimen exchanges take place through a chamber door which exposes the specimen stage when opened. Exchange time takes a few minutes. Software and interlocks protect the system against a damage and users against an injury.

IMAGE VIEWING AND CAPTURE

Because the amplified detector signal is displayed synchronously with the beam scanning, there is a correspondence between the brightness of an image point on the monitor screen and the signal detected at corresponding point on the specimen.

Magnification is the ratio of the size of the viewing monitor screen to the size of the area scanned on the specimen. Increased magnification is achieved by reducing the size of the area scanned on the specimen.

POSITIONING OF STAGE

A choice of computer-controlled high-accuracy multi-axis stages offers precision specimen manipulation and automation for overall spatial orientation on highly repetitive or extremely irregular samples.

CONTROL OF BEAMS

FIB/SEM instrument position the point of interest ideally for simultaneous ion beam cross-sectioning and electron beam viewing. Separate scan generators for each beam permit coupled or independent scan patterns and magnifications. Imaging while milling aids in defining milled features.

Immediate electron beam images of cross sections are possible without stage motion or sample transfer. Immediate high-resolution SEM imaging after FIB milling also prevents exposure of milled cross sections to atmospheric contaminants.

The ion system works only in the HiVac mode.

GAS DEPOSITION

Multiple FEI's Gas Injection System (GIS) can be installed for material deposition in conjunction with either electron or ion beam pattern definition. Electron beam-induced deposition offers the advantage of not sputtering the deposited material or implanting gallium simultaneously.

Gas Enhanced Etch

The GIS also provides Enhanced Etch™ capability for high aspect ratio drilling with minimal re deposition, as well as metal deposition, preferential etching of cross-section surfaces prior to SEM imaging, and rapid milling of TEM sections.

Up to four GIS chemistries can be installed on the instrument, depending on a system configuration. This self-contained apparatus allows the precursor material to be contained entirely within the vacuum system for simple, flexible, and safe operation.

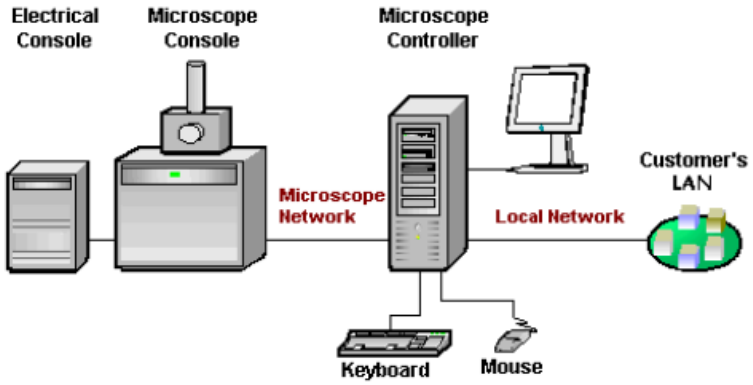
X-RAY ANALYSIS CAPABILITY

Energy Dispersive X-ray (EDX) provides elemental analysis capability for identification of surface and subsurface features. Convergence of the SEM, FIB, and EDX at short working distance allows precision "slice-and-view" cross-sectioning and chemical analysis at high resolution. Various vendor options are compatible with the instrument.

FIGURE 2-2 QUANTA 3D FEG

The standard layout is based around a dedicated microscope controller along with an electrical console to power the microscope console (vacuum, gun, column, stage etc.).

FIGURE 2-3 QUANTA 3D FEG STANDARD LAYOUT SCHEME



SOFTWARE INTERFACE ELEMENTS

The software control contains graphic applications within Windows XP™ operating environment. **xT microscope Server** starts and stops the basic microscope functions. It makes possible to open and close the **xT microscope Control** software (**UI** – user interface or sometimes **xTUI** in the dialogue boxes) which controls system functions including detection and analysis, scanning, image gathering, manipulation and output, magnification, pressure, etc.

All user account levels created via **FEI User Management** software ensure for the particular users admission to both the operating system Windows XP and the xT microscope Control software. The hierarchy of user account levels consists of the following:

- FEI Account Administrator
- FEI Supervisor Users
- FEI Microscope Users
- FEI Non-active Users

For information on Logging on and Logging off, the start-up of the system and all the features of the user interface elements see Chapters 3 and 4.

HARDWARE INTERFACE ELEMENTS

The Quanta 3D FEG system is computer controlled and as such has a **Microscope controller** which must be turned on to operate the microscope by means of the software. The power button on the microscope controller must be used to turn it on. The **Support computer** (option) can hold some other software utilities. The **switch box** switches the keyboard and the mouse to either of the two computers. The control software facilities and data are displayed graphically on the **LCD monitor** and are superimposed around and on the image (the other LCD monitor is used for optional or related programs). To control software utilities one can use a **keyboard, mouse, joystick** (option) or the **Manual User Interface** (option).

System Control Panel

The console / system power is activated by pressing the front panel **power button** located on the microscope console. This switches the sub-systems on and allows the interface and communication with the microscope controller. Most of the functions are activated via the software control. The power button green light indicates the Full Operation state, the orange one the Standby state.

FIGURE 2-4 SYSTEM CONTROL PANEL POWER BUTTON

ON

Standby

Stage Controls

The stage is software controlled (50 mm stage also manually) and can be oriented with reference to five axes: **X**, **Y**, **Z**, **Rotation** and **Tilt** (see Chapter 7).

FIGURE 2-5 HARDWARE STAGE CONTROLS
QUANTA 3D FEG 200 / 600

Final Lens Aperture Strip

The strip is made from a Mo coated Si. Automatic Aperture System (AAS) motorized software-control enables to choose the aperture most applicable to your imaging needs (see Chapter 5).

TABLE 2-1 APERTURE SIZES AND THEIR USE

No.	Standard FP 6174/55	Option FP 6174/33	Option FP 6174/37	Recommended use (related to Standard sizes)
1	1000 µm	1000 µm	1000 µm	Analytical mode, High current applications, Service Alignment
2	-	-	-	
3	50 µm	100 µm	30 µm	High current applications
4	40 µm	50 µm	30 µm	X-ray mapping of low-Z elements at low voltage
5	30 µm	40 µm	20 µm	General imaging or X-ray analysis, Dynamic experiments
6	30 µm	30 µm	15 µm	
7	20 µm	30 µm	10 µm	High resolution imaging

Caution!
The aperture holes edges cleanness is very important (see Chapter 8).

Quanta 3D FEG Vacuum System

There are following vacuum sections:

- Electron gun
- Electron column
- Ion gun
- Ion column
- Specimen chamber

In operation the electron and ion guns are always pumped to high vacuum. The specimen chamber is at the pressure required for the given state (Pump / Vent) or mode (HiVac / LoVac / ESEM).

Both Columns and Specimen Chamber sections are vented for a sample or detector exchange. In operation the Gun and Column sections are always under the high vacuum.

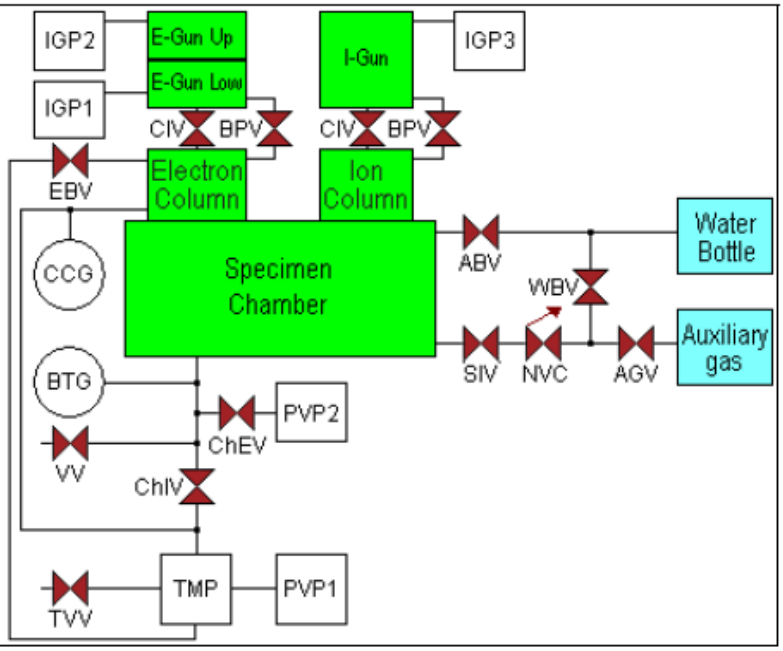
All valve and pump operations are fully automatic.

Note:
The ion beam can only be operated in HiVac. When LoVac or ESEM mode is chosen, the ion column CIV is closed.

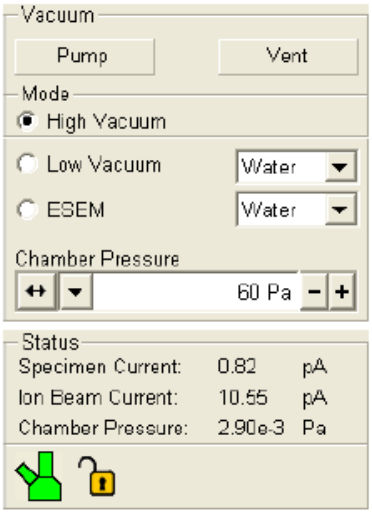
FIGURE 3-1 THE QUANTA 3D FEG VACUUM SYSTEM

Legend:

ABV . . . . Auxiliary Bypass Valve
AGV . . . . Auxiliary Gas Valve
BPV . . . . By Pass Valve
BTG . . . . BaraTron Gauge
CCG . . . . Cold Cathode Gauge
CIV . . . . . Column Isolation Valve
EBV . . . . Environmental Backing Valve
ChEV . . . Chamber Evacuation Valve
ChIV . . . . Chamber Isolation Valve
IGP . . . . . Ion Getter Pump
NVC . . . . Needle Valve Control
PVP . . . . Pre Vacuum Pump
SIV . . . . . Servo Isolation Valve
TMP . . . . Turbo Molecular Pump
TVV . . . . Turbo Venting Valve
VV . . . . . Venting Valve
WBV . . . . Water Bottle Valve



Vacuum Status



The vacuum status controls are in the **Vacuum** module. The **Pump** button starts pumping for the operating pressure and the **Vent** button starts venting for a sample or detector exchange.

In the **Status** module at the bottom of any page the actual vacuum status is represented by the colored icon, which may have three possible colors with the following meaning:

- **Green: PUMPED** to the desired vacuum mode (see below)
- **Orange: TRANSITION** between two vacuum modes (pumping / venting / purging)
- **Grey: VENTED** for sample or detector exchange

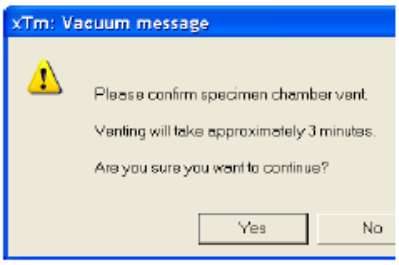
PUMP BUTTON

When the **Pump** button is clicked and the status is **Vented,** or when changing vacuum mode, the target pressure that the system pumps to depends on the selected vacuum mode. The Pump button is highlighted in and not accessible.

For the **High Vacuum** the system achieves the lowest pressure possible. For the **Low Vacuum** or **ESEM** it achieves the pressure specified in the **Vacuum** module / **Chamber Pressure** adjuster. The purge function can be defined in the **Preferences…** dialogue / **ESEM** tab (see Chapter 4).

When the **Pump** button is clicked and the status is **Transition** (venting), the venting procedure stops and the system immediately starts to pump to the actually selected vacuum mode.

VENT BUTTON

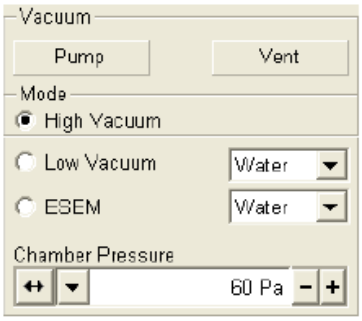


When the **Vent** button is clicked and the status is **Vacuum**, the confirmation dialogue appears. After confirmation, the system switches off the detectors voltage, high voltage supplies, vacuum pumps and uses the appropriate valves to vent the system, with the use of the dry Nitrogen brought to the **Nitrogen Inlet**.

The Vent button is highlighted and not accessible. After a specified venting time the venting valve closes and the vacuum status should indicate **Vented**. The chamber door could be opened and the button is enabled again.

When the **Vent** button is clicked and the status is **Transition** (pumping), the dialogue appears. After confirmation, the pumping procedure stops and the venting procedure starts.

When the **Vent** button is clicked and the status is **Vented**, the dialogue appears. After confirmation, the venting valves re-open for the specified venting time and then the valves close.



The **Vacuum** module / **Mode** / **High Vacuum** or **Low Vacuum** or **ESEM** radio button is used to select the instrument target operating mode when a **Pump** sequence is initiated.

PRESSURE LIMITING APERTURE (PLA) AND DETECTOR CONES

The maximum allowed specimen chamber pressure in LoVac or ESEM mode is determined by the size of the **PLA** and a gas type.

The **PLA Configuration** dialogue prompts to inform the system about the PLA size according to the figure and the application. It is used when pumping or switching to LoVac or ESEM mode. Along with a gas type, this information sets pressure limits and rates for pressure changes.

Clicking the **OK** button after selecting an appropriate pole piece radio button (cone or a detector with an integrated PLA) informs the system that it is mounted on the lens insert. The system starts pumping to the LoVac / ESEM mode. From that point on, this information is automatically used until the system is vented again, when the PLA is set to "unknown". Clicking the **Cancel** button leaves the system in its actual status mode (Pumped or Vented).

- The **Low kV Cone** (500 µm aperture) is installed in case the LFD is used for LoVac and low voltage imaging (i.e. below 5 kV) to reduce beam loss in the gas. It is used when imaging at shorter working distances (< 9 mm) and restricts the lower magnification limit.
- The **X-ray Cone** (option - 500 µm aperture) is used for EDX analysis (see Chapter 9) at a longer working distance. Samples are scanned at 10 mm working distance, which is the stage eucentric position and the collection point of the EDX detector. It is used in conjunction with the LVSED.

  The longer profile of this cone minimizes the low voltage beam dispersion and skirting of the primary beam in the gaseous environment of the chamber, allowing more electrons to interact with the specimen when focused and increasing the signal to noise ratio.
- The **GAD Cone** (option - 500 µm aperture)
- The **Heating Stage Cone** (option - 1000 µm aperture) is used with the heating stage in combination with the hook wire or LFD. It can be used without the heating stage when beam protection is desired with a larger field of view.

Lens Inserts

The cone itself or the detector with an integrated cone is possible to install to the lens insert. Run the **8 - System Configuration** alignment to determine which insert is actually used (see Chapter 6).

1. The **High Vacuum** mode insert (no apperture, no O-ring)
2. The **Standard** LoVac mode insert (3× carbon tube, O-ring) used up to 200 Pa.
3. The **ESEM** mode insert (5× apperture, O-ring) used up to 2700 Pa in conjunction with additional cone (detector).

   Gaseous detectors or the cones are pushed onto the ESEM Insert to form a gas seal. Chamber gas flows through the gas-restricting apertures found inside the insert. These apertures are made from platinum (3 pcs.) or carbon (1 pcs.). The carbon one provides a slightly broader field of view.

Note:
When no insert is mounted, imaging drifts significantly because of a component charging.

Note:
The PLA also acts as a final or objective aperture so the pressure over it is considered to be very low. Any pollution that accumulates on the aperture edge greatly affects the imaging. If astigmatism is not possible to correct, it is usually a sign that this aperture needs to be cleaned or replaced (see Chapter 8).

Caution!
Applying a cone or a detector with an integrated cone scales down the space between the top sample surface and the cone (at the particular working distance) about the value equivalent to a cone height.

TABLE 3-1 WD DECREASE WHEN USING CONES

Cone Type	Cone height [mm]
Low kV	3.5
X-Ray	8.5
BSED	3.5
GAD	5.5

HIGH VACUUM (HiVac) MODE

The high vacuum condition is common throughout the column and specimen chamber. The pressure should be below 6.10^{-2} Pa.

LOW VACUUM (LoVac) AND ESEM MODES

In these modes, the column section is under the lower pressure than the specimen chamber where the pressure ranges from 10 to 130 Pa (LoVac) or from 10 to 4000 Pa (ESEM). These modes uses water vapour from a built-in water reservoir or a gas from an auxiliary gas inlet.

When **Low Vacuum** / **ESEM** mode is entered from **ESEM** / **Low Vacuum** mode by selecting an appropriate mode radio button, nothing happens unless there is a different gas type being used for the two modes. In this case, the appropriate gas type is selected.

When **Low Vacuum** / **ESEM** mode is entered from the **High Vacuum** mode by selecting an appropriate mode radio button or from the **Vented** status, the system prompts an user with the PLA Configuration dialogue (this happens only for the first time after a particular Vent procedure).

Pressure

The **Chamber Pressure** adjuster is used to set and display the target chamber pressure. Pascal, Torr or Millibar units are available and can be selected in the **Preferences...** dialogue / **Units** tab (see Chapter 4).

When the system is in LoVac or ESEM mode and the **Chamber Pressure** value is changed, the pressure automatically changes to the new value. When the system is in any other state and the chamber pressure value is changed, the new value is used as the target pressure when the system starts pumping to a Low Vacuum or ESEM mode again.

The actual specimen chamber pressure is displayed in the **Status** module / **Chamber Pressure:** field.

Using Gas

ESEM and Low Vacuum modes allow an user to image samples in a gaseous atmosphere, which can be selected in the drop down list box:

- the **Water** vapour from a built-in water reservoir located in the back part of the microscope console,
  Note:
  On occasion the water reservoir needs to be filled (see Chapter 8).
- the gaseous environment supplied by an user via the **Auxiliary gas** inlet placed on the back of the console.
  Caution!
  Maximum overpressure for the Auxiliary gas and Nitrogen inlets is 10 kPa (0.1 bar). The **Nitrogen** inlet is used only for venting the chamber with air or the nitrogen preferably.

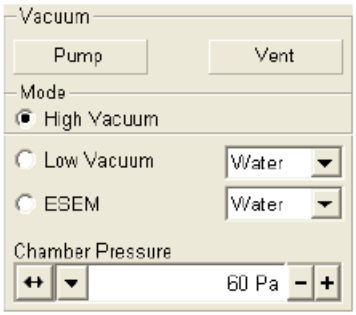
When using a particular pressure limiting aperture, there are pressure limits for different gasses.

TABLE 3-2 MAXIMAL CHAMBER PRESSURE [PA (TORR)] UNDER DIFFERENT GASEOUS ENVIRONMENT

Working Gas	500 µm Aperture	1000 µm Aperture
Water - H_2O	2 700 (20)	750 (5.5)
Nitrogen - N_2	4 000 (30)	750 (5.5)
Air		
Carbon Dioxide - CO_2		
Nitrogen Dioxide - NO_2		
Helium - He	2 000 (15)	500 (4)
70% He + 30% H_2	1 500 (12)	400 (3)
Argon - Ar **)	1 000 (7)	200 (1.5)
C_xH_y *)	4 000 (30)	750 (5.5)

Note:
*) Combustible gases (acetylene for instance) must always be used with respect to safety issues.
**) The argon use should be minimized to a short time, because the IGP's are not optimized for pumping of it at all.

Purging

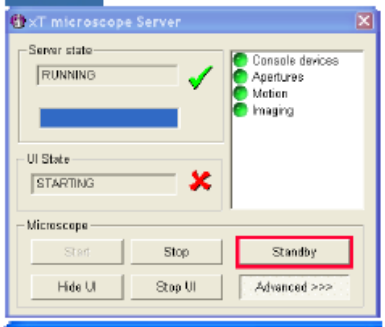


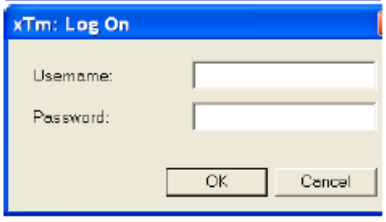
During this procedure the specimen chamber is automatically pumped down to a lower pressure to remove the old gas, then it is flooded with the new one (selected in the Vacuum / Mode module) to a higher pressure. This takes place several times, until the old gas is removed and the chamber is mostly filled with the new gas. This is applied when the system is:

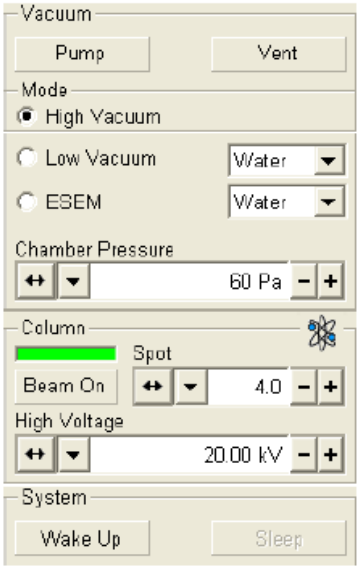
- pumping to the LoVac / ESEM mode from the vented status.
- in the LoVac / ESEM mode and the gas type is changed.
- in the LoVac / ESEM mode and the **Purge** button is pressed.

The purging can be set up, started and terminated in the **Preferences...** dialogue / **ESEM** tab (see Chapter 4).

Note:
This procedure can take several minutes, according to Preferences setting. Wait until Vacuum status indicates **Vacuum**, because detectors do not start operation till desired pressure is reached.







There are several system states:

1. **Complete Shutdown** – service and emergency reasons
2. **Standby** – when not using the system for a longer period
3. **Overnight** – when not using the system overnight
4. **Full Operation** – when working

TABLE 3-3 STARTUP PROCEDURE GENERALLY

System State	Action
from Complete Shutdown to Standby	**1)** Connect the power cord to the microscope console, compressed air, water and the nitrogen inlets. Interlock prevents the vacuum system from operating if a compressed air is not connected. **2)** Start the electron column IGP pumps with the **5 - Emitter Startup** and the ion column IGP pump with the **100 - ION: Source Control** alignment (see Chapter 6). **Note:** If this is not possible, call the service!
from Standby to Overnight	**3)** Press the **power** button on the microscope front control panel. The orange light changes to the green one. **4)** Switch on the PC. The operating system [(a)] Windows XP loads and displays the appropriate icons on the monitor desktop. **5)** Double-click the **xT microscope Server** icon to start the software (all seeming LED's should be green). **6)** Click the **Start** icon to start the server. Wait until all dialogues are fully functional. **7.** Click the **Start UI** button to start the **xT microscope Control** software. The main window appears behind the **XTUI Log On** dialogue. **8.** Enter your **Username** and a **Password** [(a)]. **9.** Switch on the Emitter with the **5 - Emitter Startup** alignment (see Chapter 6).
from Overnight to Full Operation	**10.** Select the **Vacuum** mode and pump the system. Wait for the **Pumped** status. **11.** Click the **Column** module / **Beam On** button (must be yellow) to start the selected beam only or the **System** module / **Wake Up**[(b)] button to start both beams. A **Column** module / **Source** progress bar indicates the selected beam start.
	See Chapter 5 to set desired imaging conditions.

Note:
When switching the console on (starting from the Standby mode), the SEM aperture strip becomes heated. It may take several (5 - 10) minutes before the strip is at the proper temperature. Do not make any adjustments during this interval (aperture, gun or lens alignment)! The microscope could become misaligned when the strip reaches the operational temperature.

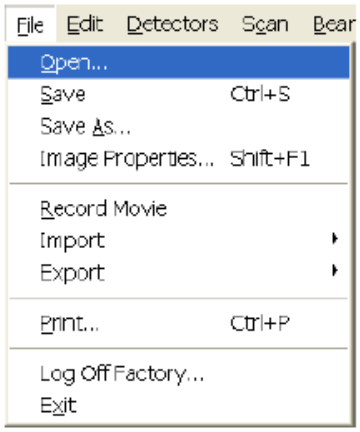
Note:

[a] Once you have your **FEI Microscope user** (or **Supervisor**) **account** set up via **FEI User management** software by **FEI Account Administrator** (see Chapter 4), you can use your name and password to access both Windows XP system and the **xT microscope Control** software. (Take note of the case sensitive passwords necessary at Windows XP and xT microscope Control server Log On points. A password is advisable for logging on to protect individual settings and results.)

[b] Usually, the Quanta 3D FEG remains on with the vacuum system in operation, but typically the ion emitter and accelerating voltages for both columns remain off. The system starts with the setting in use when the **xT microscope Control** software was closed. This allows quick resumption of daily operation.

TABLE 3-4 SHUT DOWN PROCEDURE GENERALLY

System State	Action
from Full Operation to Overnight	**1)** Click the **System** module / **Sleep** button to switch off both beams and to stop the ion source. **2)** Select the **Stage** menu / **Tilt 0°** (Ctrl + E). Click the **Vacuum** module / **Vent** button. Wait for the **Vented** status. Remove your sample if needed and remove the Cooling stage if installed. If you want to shutdown the microscope, bypass steps 3 - 7. **3)** Click the **Vacuum** module / **Pump** [c] button to pump to the High Vacuum. **4)** Select [a] the **File** menu / **Log Off...** to log off the present user and to provide the **Log On** dialogue for entering another one.
from Overnight to Standby [b]	**5.** Click the **Standby** button to stop the **xT microscope Server** software services. **Note:** If the system is left in the Pumped status, the pumps keep the vacuum all the time (HiVac or the pressure 60 Pa for the LoVac / ESEM mode). If it is left in the Vented status or when the Standby button is clicked during the Transition status the pumps do not keep the vacuum. **6.** Exit the **xT microscope Server** (right-clicking) and **Windows XP**. **7.** Switch Off the PC and the monitor.
from Standby to Complete Shutdown	**8.** Switch off the Emitter (see Chapter 6). [e] **9.** Click the **Standby** button to stop the **xT microscope Server** and to switch off the console. The **power** button on the microscope front control panel changes the green light to the orange one. **10.** Exit **xT microscope Server** software (right-clicking) and **Windows XP**. **11.** Switch Off the PC and the monitor.
Service [d]	**12.** Disconnect the power cord and any other optional input / output if used.



Note:

a) Waiting for a new user leaves the status of the **xT microscope Control** software non-operational and only the **xT microscope Server** software is active. Therefore changing an user does not require Logging off / Logging on at Windows XP level, but just restarting the UI level.

b) The power plug should not be disconnected. The system can be left in this state if electrical power is supplied to the instrument because the pumps are running and pumping the column.

c) It is strongly recommended to always leave the chamber in HiVac mode when not being used. When the sample chamber is left in the LoVac / ESEM mode, water vapour is likely to accumulate in it, PVP lifetime decreases and the water reservoir or gas cylinder empties prematurely.

d) Disconnecting the power cord brings the system to the non-powered state, where the vacuum in the instrument is no longer supported by running pumps. All valves are closed, and the electron column and specimen chamber areas are vented gently.
This should only be carried out by a FEI service engineer. Normally it is used for a system transportation or for service actions, like repair to essential systems (electrical and air supplies).

WARNING!
The electron column ion getter pumps are equipped with a backup power supply. Even when power cable is disconnected, the electron column IGP's are still ON!

(e) Switching off the console when **Emitter is On** is not optimal and may deteriorate the filament lifetime.

POWER OFF

Take sufficient measures to avoid power failures as much as possible. If it occurs while the instrument is completely operational, the microscope comes down to a safe state and the following happens:

- The HV is switched off abruptly.
- Both electron and ion emissions are switched off.
- The specimen chamber vents gently, automatically.
- The column isolating valves close to save the vacuum in the gun areas.

  Note:
  Electron gun IGP's are supported by the battery unit for several hours to keep sufficient vacuum for the source to operate.
- The momentary adjustments of all system parameters (accelerating voltage, magnification, stage positions) are lost if they were not saved.

Note:
If the power failures occur occasionally it is recommended to use the microscope Uninterruptible Power Supply (UPS).

The Emergency Off

is similar to that which would happen after a MAINS power off. Here are several possibilities how to quickly switch off the electrical power completely in case of emergency:

1. Push the red **EMERGENCY** (EMO) button (option - see the Safety Manual).

If the button is not installed proceed as follows:

2. Switch off the breaker switch labeled **MAINS S1** at the cabinet back, which is placed at the very right side in the row.

FIGURE 3-2 MAINS SWITCH BOARD

If this is not easily accessible:

3. Turn off the mains wall switch (if present), and / or disconnect the mains plug from the mains socket.

C a u t i o n !
If the Startup procedure fails (see above), contact the FEI Service Engineer.

MOUNTING SPECIMEN TO THE HOLDER

Wafers and PGA devices have individual sample-mounting procedures. If you are using a wafer piece or other sample, attach the specimen to the specimen holder using any suitable SEM vacuum-quality adhesive, preferably carbon paint. The specimen must be electrically grounded to the sample holder to minimize specimen charging. If you are using a vice mechanism or double-sided tape, make sure the specimen is conductively attached to the holder.

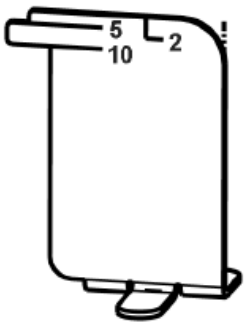
Note:
The sample holder is not directly grounded to the chamber ground because it is connected to the BNC feed on the chamber door. This allows to measure the specimen current.

Caution!
Store samples and sample holders in a dry and dust free environment. Dust on samples can get drawn into the electron column, degrading imaging and requiring an FEI Customer Service.

INSERTING / EXCHANGING SPECIMEN AND / OR DETECTOR

It is assumed, that the microscope is in the Full operation state (see Chapter 3).

1. Click the **Vacuum** module / **Vent** button. The confirmation dialogue appears.
   After a High Voltage switch off, the vacuum system switches off the pumps and opens the appropriate valves to vent the system. After a specified venting time the venting valve will close.
   Note:
   If the venting valve closes before the chamber is at the atmospheric pressure (the door is not possible to open), click the Vent button once more to open it again.
   If you vent the system in order to change a detector, wait until the **Status** module vacuum icon (chamber part) is grey. Otherwise there is a risk of a detector assessment malfunction, and as a result the PLA (see below) is not recognized by the system.
2. When vented, open the specimen chamber and, using lint-free gloves or tweezers, place a specimen into the specimen holder. Secure the specimen stub with an appropriate hex-wrench unless a spring-clip holder has been used.
3. Install any additional detector if it is not already done (see below).
4. Adjust the Eucentric Position (see Chapter 7).
5. Close the specimen chamber door.



OPERATION PRE-CHECK

To ensure correct operation in any Vacuum mode, check the following list before continuing. After obtaining a preliminary imaging, you can then experiment with your settings.

TABLE 5-1 QUANTA 3D FEG SETUP CONDITIONS

Adjustment	E-Beam Setting
Vacuum mode (Lens insert)	**HiVac:** conductive samples **LoVac:** nonconductive, mixed or contaminating samples **ESEM:** wet samples (use H_2O gas medium)
Accelerating Voltage	Select voltage relative to specimen type: - low voltage for surface imaging, beam-sensitive samples and slightly charging samples - high voltage for conductors, high resolution, compound info (BSE, X-ray) For example: - biological sample High Voltage = (1–10) kV - metal sample High Voltage = (10–30) kV
Spot size	**HiVac** and **LoVac:** 5 or 6 **ESEM:** 6
Pressure	**HiVac:** the lowest **LoVac:** 60 Pa (0.5 Torr) **ESEM:** 600 Pa (3.7 Torr)
Scan rate	**HiVac:** fast (dwell time about 0.1 - 0.3 µs) **LoVac** and **ESEM:** slow (dwell time about 1 - 3 µs)
Working Distance	Set the highest specimen point to approximately 10 mm (yellow mark in an optical quad) and press Ctrl + F to set WD to 10 mm.
Magnification	Set to lowest – from 50× to 200×
Standard Detector	**HiVac:** ETD (SE) **LoVac:** LVSED **ESEM:** GSED
Filtering	**HiVac:** Average (4 frames for fast scans) **LoVac** and **ESEM:** Live
Contrast and Brightness	With contrast at minimum value adjust brightness to just show a change in intensity to the screen. Increase the contrast to produce a reasonable imaging. Increases in brightness and decreases in contrast produce softer imaging and vice versa.